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ADVANCED ANALYTICAL METHODOLOGY FOR MOBILITY FUELS AND LUBRICANTS APPLICATIONS

INTERIM REPORT
BFLRF No. 273

By

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A review is given of state-of-the-art analytical chemical methodologies and instrumentation to provide timely evaluation of the quality of fuels and lubricants. An assessment is also provided for the possible use of these techniques under battlefield and near-battlefield conditions by marginally trained petroleum supply technician.

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EXECUTIVE SUMMARY

Problems and Objectives: The U.S. military experienced equipment failures due to misformulated or inappropriately chosen fuels and lubricants. It is the purpose of this report to summarize present and near-future methodologies and technologies to alleviate these problems through the application of appropriate analytical techniques and methods to provide accurate and timely analyses to assess the quality of fuels and lubricants.

Importance of Project and Military Impact: Through knowledge gained as a result of the studies outlined in this report, the frequency and severity of fuels- and lubricants-related equipment failures may be minimized, thus, increasing the useful life of the effected machinery at a minimum cost.

Technical Approach: Results of an extensive computer base literature survey were combined with the consensus of experts in this field to arrive at conclusion and recommendations outlined in the report.

Accomplishments: This report includes an introduction, a grossly abbreviated discussion of the applicable methodologies, the classification of these methodologies, literature references, and definitions. Additionally, the report summarizes the challenges that the subject matter poses for chemists, for computer scientists, and equipment manufacturers to modify existing systems, or, when needed, to develop new systems and methodologies.

FOREWORD/ACKNOWLEDGMENTS

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I. INTRODUCTION

The drive to make modern military equipment more proficient has resulted in the use of more complex and expensive components with critical and exacting requirements. A major requirement for the operation and maintenance of such equipment is an adequate supply of the proper fuel and lubricant. To assure reliable and rapid response to problems related to the quality of mobility fuels and lubricants, state-of-the-art test instrumentation is essential. Such analytical instruments will provide the field commanders with the necessary data about the usability of POL products and will result in reasoned recommendations in the shortest time possible.

Thus, there is a strong need to develop analytical chemical equipment capable of ensuring quality control of petroleum products in or near battlefield conditions. This equipment, which should be transportable and, perhaps, portable, will permit the timely assessment of the quality of fuels and lubricants at Army facilities. The use of this equipment would identify specific POL products and determine their compliance with the respective specifications. If needed, recommendations for corrective actions would be provided by the equipment. Such systems should also identify host nation, commercial, or unknown source POL products that may be used in lieu of proven, fully acceptable (QPL) products either as an alternative or as an emergency fuel or lubricant.

There are two major areas of testing of products. The "Petroleum Quality Analysis System" (PQAS) describes quality assurance and quality conformance testing of products prior to delivery to assess if the supplied products are acceptable for field use. In MIL-HDBK-200G (1)*, requirements are given for POL product quality assurance already in field stock.

The fast growing relatively recent science on artificial intelligence (2) and the development of expert systems (3,4) are expected to provide substantial improvements in advanced analytical chemistry, including those that are used by the military.

* Underscored numbers in parentheses refer to the list of references at the end of this report.

This report includes an introduction, a grossly abbreviated discussion of the applicable methodologies, the classification of these methodologies, literature references, and definitions. Additionally, the report summarizes the challenges that the subject matter poses for chemists, for computer scientists, and equipment manufacturers to modify existing systems, or, when needed, to develop new systems and methodologies.

II. MILITARY STANDARDIZATION HANDBOOK MIL-HDBK-200G AND QUALITY ASSURANCE/QUALITY CONFORMANCE TESTING REQUIREMENTS

This publication is a "quality surveillance handbook for fuels, lubricants and related products." It is considered as the present foundation and starting point from which future trends and requirements may be developed. The book defines the specification and use limits for the various products and the acceptable analytical procedures that must be implemented by "properly trained personnel ... to protect original product quality." The handbook classifies the tests into four categories, corresponding to four levels of effort.

In **TABLE 1,** a summary of MIL-HDBK-200G specified measurements is given for aviation and automotive gasolines, for turbine, diesel, and burner fuels, and for lubricating oils. The table also includes the requirements defined for the four levels of analytical effort included in MIL-HDBK-200G. Significances of the various tests (5) are briefly discussed in the Appendix.

Type "A" tests (not included in MIL-HDBK-200G) are the complete set of quality assurance, quality conformance, or specification inspection tests. Requirements for specific products are described in the various specifications, for example:

- MIL-G-3056 — Gasoline, Automotive, Combat
- MIL-T-5624N — Turbine Fuel, Aviation, Grades JP-4 and JP-5 and JP-5/JP-8 ST Special Test Fuel
- MIL-T-83133C — Turbine Fuel, Aviation, Kerosene Types, Grades JP-8 (NATO F-34) and NATO F-35
- MIL-F-46162C — Fuel, Diesel, Referee Grade
- VV-F-800D — Fuel Oil, Diesel
- VV-F-815 — Fuel Oil, Burner

TABLE 1. Summary of Required Tests in MIL-HDBK-200G

Property	POL Application by Test Category					
	GA	G	TF	DF	BF	LO
Acid Number	--	--	--	2	--	--
Accelerated Stability	--	--	--	2	--	--
Appearance	--	123C	--	123C	--	12C
Ash	--	--	--	--	2	2
Basic Sediment & Water	--	--	--	--	123C	--
Carbon Residue	--	--	--	12	1	2
Cetane Index	--	--	--	2	--	--
Cloud Point	--	--	--	2	--	--
Color	123C	123C	123C	123C	--	12C
Conductivity	--	--	123	--	--	--
Corrosion	123	23	123	2	--	2
Distillation	123	123	123	12	--	--
Existent Gum	12	--	123	--	--	--
Filtration Time	--	--	123	--	--	--
Flash Point	--	--	123C	123C	123C	12C
Foam	--	--	--	--	--	2
Freezing Point	--	--	123	--	--	--
Fuel System Icing Inhibitor	--	--	123	--	--	--
Gravity	123C	123C	123C	123C	--	12C
Gum, Unwashed	--	23	--	--	--	--
Knock Rating	--	12	--	--	--	--
Lead Corrosion	--	--	--	--	--	2
Lead	12	3	123	--	--	--
Lean Mixture Rating	123	--	--	--	--	--
Metals	--	--	--	--	--	2
Neutralization Number	--	--	--	--	--	2
Oxidation Stability	--	2	--	--	--	2
Particulates	--	--	--	12	--	--
Peroxides	--	--	2	--	--	--
Potential Gum	2	--	--	--	--	--
Pour Point	--	--	--	2	2	2
Precipitation No.	--	--	--	--	--	2
Protection	--	--	--	--	--	2
Rich Mixture Rating	12	--	--	--	--	--
Saponification No.	--	--	--	--	2	2
Sediment (Ext)	--	--	--	--	2	--
Separation	--	--	--	--	--	12C
Solids	123	--	123	--	--	12
Sulfur	--	--	2	--	--	--
Thermal Stability	--	--	2	--	--	--

TABLE 1. Summary of Required Tests in MIL-HDBK-200G (Cont'd)

Property	POL Application by Test Category					
	GA	G	TF	DF	BF	LO
Vapor Pressure	12	12	123	--	--	--
Viscosity	--	--	--	2	12	12C
Water Separation Index	--	--	123	--	--	--
Water Reaction	123	--	123	--	--	--
Water & Sediment	--	--	--	2	--	--
Water	--	--	--	--	--	12C
Water & Solids	123C	123C	123C	--	--	--
Water Tolerance	--	123	--	--	--	--

Notes:

Products:
GA = Gasoline, aviation
G = Gasoline, automotive
TF = Turbine fuel
DF = Diesel fuel
BF = Burner fuel
LO = Lubricating oil

Test Categories:
1 = Type B-1 test
2 = Type B-2 test
3 = Type B-3 test
C = Type C test

Type "B-1" tests demand partial analysis comprising the checking of principal characteristics most likely to have been affected in the course of moving the product. Type "B-2" tests specify partial analysis to verify characteristics susceptible to deterioration because of age. Type "B-3" tests describe requirements for partial analysis for contamination; in particular, for controlling the reinjection of pipeline interface products. Type "C" tests include the execution of basic (minimal) analyses to ascertain gross acceptability criteria. Type "C" tests consist of measurements such as specific gravity, color, and appearance, including visible sediment and water.

According to a recent concept statement (6) (an initial "organizational and operational, or O&O, plan") by the Quartermaster School for automated petroleum testing capabilities in the battlefield environment, there are three testing systems for three areas of operation:

- Tactical testing at forward areas, equipment should be man-portable and be limited to aid fast and simple go/no-go decisions.

- Operational testing allows the use of a limited number of tests that would be performed on sophisticated, transportable equipment. Operators of this equipment would be "school-trained petroleum laboratory specialists (who) will perform B/C level tests, interpret results, and provide expert opinion on petroleum products." Due to increased capability and sophistication, such laboratories would be able to provide improved precision and reliability of data and recommendations. The analytical equipment would be housed in trailers or transportable modules.

- Theater level testing would provide the most comprehensive analyses, interpretation, and recommendations for the commanders. In future scenarios, analyses of petroleum products would be performed using a battery of state-of-the-art equipment, interfaced with a computer-based expert system. This level of testing would be performed in portable shelters.

III. METHODOLOGIES

In the following section, brief descriptions will be given on several state-of-the-art instrumental analytical chemical techniques. Fully developed, these instruments may be adopted to provide faster acquisition of higher quality and more reliable measurements in the hands of moderately trained personnel. Proper selection of these instruments should result in development of methodologies that, with the inclusion of appropriate computer programs, would allow essentially automated analysis of U.S. Army fuels and lubricants.

Selection of tests for MIL-HDBK-200G was moderated by the availability of simple, traditional procedures that were adaptable for military applications. In an integrated future POL laboratory system, some of the traditional test procedures are expected to be retained, while others may be replaced by newer methodologies that would provide improved speed, reliability, and more

meaningful input about the usefulness and limitations of such products to the military commanders.

As shown in the following section, single measurements may yield data that either directly measure desired property values, or the acquired data may be correlated with several specification-type test results, thus reducing the number of measurements required to guarantee quality assurance.

Engine-performance tests are not included in this report, as those are considered impractical due to time restraints under battlefield conditions.

The indicated analytical time requirements reflect present needs and are expected to be reduced in future advanced systems. It should be noted that the order of the discussion does not indicate the order of importance of the analytical method.

Some of the instrumentation that will be discussed below, at present, require special handling and environment. The equipment may need a vibration-free platform, cryogenic cooling of detectors, etc. Such restrictions, however, may be circumvented by future development.

1. **Elemental analysis** of fuels and lubricants provides determination of carbon, hydrogen, oxygen, nitrogen, and sulfur, as well as a host of other elements that may be part of the system, e.g., calcium, iron, lead, zinc, etc. Using presently available automated equipment, performing such analyses would require several instruments, such as C, H, N, S analyzer, nuclear magnetic resonance (NMR) based hydrogen analyzer, energy-dispersive X-ray spectrometer (if cryogenic cooling of the detector is possible), atomic absorption, emission spectrometers, etc.

   Application of these technologies yields chemical compositional data that may give important input to determine if a fuel or lubricant is proper for a given application.

The NMR-based hydrogen analyzer and inductively coupled plasma (ICP) elemental analyzer are expected to be usable at **operational level of testing**. The other equipment, due to their high sensitivities, are expected to be used only at **theater level applications**.

Results of elemental analysis may provide information about

- Composition
- Heat of combustion
- Aromatic hydrocarbon content
- Tendencies toward smoking
- Tendencies to cause wear and form deposits
- Presence and quantity (qualitative and quantitative analysis) of certain additives.

Time requirement: 0.25 to 1.0 hour.

2. **Fourier transform infrared spectroscopy (FTIR)** (7-13) reveals molecular bonding and is used for functional group analysis of fuels and lubricants. When compared to spectra of standard (calibration) material, this well-established technique is useful to provide powerful chemical compositional data to:

   - Provide rapid fingerprinting and initial characterization of samples
   - Identify (authenticate) samples
   - Determine quality assurance of QPL products
   - Determine alkyl nitrates (cetane number, i.e., ignition improvers) in diesel fuels (ASTM D 4046) (14)
   - Predict oxidation
   - Estimate depositing tendencies
   - Determine degree of oxidation and nitration during engine operation
   - Determine additive concentration and depletion
   - Detect and measure contamination in fuels and lubricants
   - Evaluate corrosion and wear tendencies

- Detect and estimate aromatic hydrocarbon concentrations and their substitution; these results may also provide input to the estimation of cetane and octane numbers
- Relate aromatic hydrocarbon concentration to elastomer compatibility

It is expected that with appropriate modifications, such systems may be used in **operational or higher level testing.**

Time requirement: 0.25 to 0.30 hour per sample.

3. **Near-infrared (NIR) spectroscopy** (15) detects bulk fuel properties that relate to average molecular features. The method is not applicable to the analysis of most additives because they are present in trace quantities only. A laptop computer-aided prototype version of this instrument, operating in the 700- to 1100-nm region of wavelengths, is easily man-portable. At present, such an instrument is under development to quickly determine various quality parameters of gasolines. Correlations are sought to calculate from NIR data several properties, including

- hydrocarbon-type analysis
- bromine number
- lead content
- sulfur content
- gravity.
- octane number
- cetane number
- freeze point
- vapor pressure

It should be noted that the methods have to be recalibrated for each of the various types of products, such as those gasolines that are leaded, unleaded, or if they contain ethers or alcohols.

Since NIR detects only harmonics of fundamental IR frequencies, it is possible that substantial improvements in specificity and sensitivity may be achieved by utilizing fundamental absorption bands, i.e., "normal" IR or FTIR. The advantage of NIR over FTIR

is that NIR may use less expensive and less sophisticated detectors and optical systems and materials.

If development of this technology will mature to the expected heights, it may be used in **tactical or higher level testing.**

Time requirement: 0.1 to 0.25 hour per sample.

4. **Raman spectroscopy** (16-20), while a mature science related to infrared spectroscopy, is now under investigation for the analysis of aviation turbine fuels using approximately 1 mL of sample. It is expected that Raman spectroscopy may be used to evaluate:

   - Overall fuel composition (e.g., aromatic to aliphatic hydrocarbon ratio) and
   - Specific additive concentrations, e.g., phenolic antioxidant, ethylene glycol derived icing inhibitor.

   Traditional weakness of Raman spectroscopy is that it requires that the samples be in true solution, i.e., the sample may not contain particulate or colloidal components that scatter **visible** light.

   With appropriate development, this technology may be used at **operational or higher level testing.**

   Time requirement: 0.10 to 0.25 hour per sample.

5. **Ultraviolet (UV) and visible (VIS)** (21-22) spectroscopy has been correlated with property requirements, e.g., to estimate color, aromatic ring-carbon content and degree of fuel degradation, etc., in mobility fuels. Further refinement of these methods may be possible by using state-of-the-art equipment and calibration to correlate with more quantitatively established methods, e.g., ^{13}C-NMR.

Some of the UV/VIS spectroscopic applications are:

- Analysis of naphthalene hydrocarbons in aviation turbine fuels, as described in ASTM D 1840 (23)
- Determination of fuels' tendencies toward smoking
- Detection of contamination by particulate matter in liquids
- Combustion characterization
- Determination of pyrrole content in fuels.

With appropriate development, this technology may be used at **operational or higher level testing.**

Time requirement: 0.25 to 0.5 hour per sample.

6. **Fourier transform nuclear magnetic resonance (FT-NMR) spectroscopy** (24-26), tuned to ^{1}H or ^{13}C resonances, may determine the presence and concentration of various carbon-hydrogen bond environments (atomic configurations in molecules). NMR may also be tuned to isotopes of a host of other elements (e.g., ^{11}B, ^{14}N, ^{17}O, ^{19}F, ^{29}Si, ^{31}P) to provide information about the atomic bonding environment of those species. NMR is a powerful tool to provide insight to the chemical composition of the sample.

FT-NMR measurements can provide:

- Determination of hydrogen content in hydrocarbons
- Identification and analysis of certain additives
- Identification of certain brake fluids, antifreeze compositions, and some fire-resistant hydraulic fluids
- Aromatic hydrocarbon concentrations, including substitution pattern of the aromatic ring
- Aromatic ring-carbon contents
- Hydrocarbon chain branching to estimate octane and cetane numbers, cloud point, pour point, etc.
- Presence and molecular environment of adjacent heteroatoms.

Although broad-band NMR, using a permanent magnet, is a relatively simple and rugged instrument, the high-resolution instruments require very stable working platforms and should have cryogenic environments for their detectors. Such requirements may place these high-resolution systems at a disadvantage under battlefield conditions.

The broad-band instrument may be used in **operational or higher levels of testing**. High-resolution NMR may be used only if stable instrument platforms and cryogenics (liquid helium or nitrogen) are available.

Time requirement: ^{1}H-NMR: 0.5 to 1 hour per sample
^{13}C-NMR: 1 hour to overnight per sample.

7. **Thermal analysis (TA)** (27-29) modes of thermogravimetry (TGA) and differential scanning calorimetry (DSC) may be used to correlate the obtained data with the thermal and oxidative stabilities of fuels and lubricants. Literature references include

   - Measurement of ash and residue content in lubricants
   - Measurement of soot content in used oils caused by diesel fuels
   - Determination of evaporation rate of gas turbine oils and other products
   - Determination of the induction period of oxidation of fuels and lubricants by DSC. Results of this procedure relate to thermal oxidation susceptibility of fuels and lubricants
   - Determination of specific heat of aircraft turbine lubricants by thermal analysis, ASTM D 3947. (30)

Thermal analytical instrumentation has been successfully combined with a variety of other techniques to provide a more complete elucidation of certain reactions. Examples include combining TA with high-performance liquid chromatography, infrared and Fourier transform infrared spectroscopies, atomic absorption spectroscopy, gas chromatography, mass spectroscopy, and combined gas chromatography-mass spectroscopy.

If development of this technology and correlation of data with performance parameters mature to the expected levels, it may be used in **theater level testing.**

Time requirement: 0.5 to 4 hours per sample.

8. **High-resolution capillary gas chromatography (HRC-GC)** (31-35) is one of the most useful tools that may be used to determine composition of complex mixtures if proper reference compounds and calibration are used.

The following items represent a brief description of the predominant applications of HRC-GC:

- Paraffin-olefin-naphthene-aromatic (PONA) hydrocarbon-type analysis of gasolines has been accomplished, but the same analysis for turbine and diesel fuels has not yet been established
- Oil base stock identification
- Simulated distillation to determine the boiling point distribution of mobility fuels and lubricants up to C_{100}-hydrocarbons (bp to 720°C) has been established. An ASTM procedure for the determination of a restricted boiling range of petroleum fractions by GC is given in ASTM D 2887. (34)
- ASTM D 3524 (35) describes a GC method to determine diesel fuel diluent in engine oils.
- ASTM D 3525 (36) measures gasoline diluent in used engine oils
- ASTM D 4815 (37) measures oxygenates (ethers and C_1 to C_4 alcohols) in gasoline.

A data base is under development to correlate the results of this method to several pertinent and specification type requirements, e.g.:

- Classification of fuel (gasoline, jet fuel, diesel fuel, blends of products, etc.)
- Flash point
- Viscosity
- Gravity
- Octane number

- Cetane index
- Vapor pressure.

With appropriate development, this technology may be used at **operational or higher level testing.**

Time requirement: 1.5 to 2 hours per sample.

9. **Gas chromatography combined with mass spectroscopy** (GC/MS) (38-40) enjoys wide acceptance because inclusion of MS made the powerful GC method even more authoritative for the compositional analysis of samples. At present, the disadvantage of GC/MS is that it requires the input of highly trained professionals capable of extracting the most information from expensive equipment and interpreting the complex results. The foundation has already been laid, however, for computer-assisted interpretation. Although such systems are commercially available, they still require elucidation by highly trained professionals. The methodology exists or has the potential of determining:

- Analysis of hydrocarbon types in middle distillates [ASTM D 2425 (41)];
- Hydrocarbon class and limited identification and measurement of individual compounds in fuels. Such data may be used as input to establish or determine such performance parameters as:

  - Depositing tendencies
  - Wear tendencies
  - Estimated octane and cetane numbers of fuels
  - Estimated cetane index of fuels
  - Estimated flash point of fuels
  - Estimated heat of combustion of fuels (when data are combined with the hydrogen content of the product).

With appropriate development, this technology may be used at **operational or higher level testing.**

Time requirement: 1.5 to 2 hours per sample plus, at present, 24 to 48 hours for interpretation of results for each sample.

10. **GC/FTIR/MS** combination methodology provides improved qualitative and quantitative analytical chemical characterization of mobility fuels and lubricants by identifying specific compounds with functional groups of interest. The method will ultimately be capable of identifying, for example, the specific carbonyl compounds that are formed during the degradation of specific fuels. Such investigation should provide direct proof of the existence of certain intermediates and products of fuel degradation. These intermediates may, in turn, lead to the control of desired and undesired reactions.

    Time requirement: 1.5 to 2 hours per sample plus, at present, 24 to 48 hours for interpretation of results for each sample.

    With appropriate development, this technology may be used at **theater level testing**.

11. **High-performance liquid chromatography (HPLC)** (42-59) has several major forms, including normal and reverse phase liquid chromatography, molecular size exclusion or gel permeation chromatography (GPC), ion chromatography (IC), etc. Unlike gas chromatography, separation of mixtures is not limited by sample volatility. Interpretation of results of HPLC and any other chromatographic technique should be tempered by the possibility that irreversible adsorption of certain components may prevent their eventual detection.

    The various HPLC methods may be used for compositional analysis of samples and providing preseparation of complex mixtures prior to detailed subsequent analyses. Accomplishments in this area include:

    - Hydrocarbon-type analysis of diesel fuels
    - Qualitative and quantitative analysis of polynuclear aromatic hydrocarbons
    - Estimation of molecular weight range and molecular weight change of fuels, lubricants, and additives

- Separation and isolation of product mixtures according to their polarity and solvated molecular size
- Necessary preliminary separation of components from mixtures for subsequent analysis, e.g., separation of additives from basestocks of fuels and lubricants
- Quantitative paraffin-olefin-naphthene-aromatic (PONA) hydrocarbon-type analysis of all mobility fuels is under development using a dielectric constant detector
- Quantitative determination of products of oxidation in fuels and lubricants
- Detection and analysis of certain additives, e.g., ignition improvers and lubricity improvers in fuels.

With appropriate development, this technology may be used at **theater level testing.**

Time requirement: 0.5 to 2 hours per sample.

12. **HPLC/^{1}H-NMR** (60) method is under development to predict various significant hydrocarbon fuel properties from ^{1}H-NMR data. In this procedure, the fuel is separated into hydrocarbon classes that are characterized on-line by proton nuclear magnetic resonance spectrometry.

Using mainly model compounds and also several middle distillate fuels, multivariate regression of data resulted in establishing equations that allow the empirical estimation of several physical properties of the samples. Preliminary calculated property constants show good correlation coefficients for:

- cetane & octane numbers
- boiling point
- freezing point
- flash point
- density
- refractive index
- specific dispersion
- surface tension
- critical pressure
- critical temperature
- critical volume
- heat capacity
- heat of vaporization
- heat of formation
- free energy of formation
- heat of combustion

Additional correlation studies are continuing to verify the validity of the methodology for the analysis of full boiling range (operational) hydrocarbon fuels.

If development of this technology will mature to the expected heights, it may be used in **theater level testing.**

13. **HPLC/MS** combination provides exceptional chemical compositional results, as compounds that are quantitatively separated by HPLC may be specifically identified by MS.

    If development of this technology will mature to the expected heights, it may be used in **theater level testing.**

    Time requirement: 1.5 to 2 hours per sample plus, at present, 24 to 48 hours for interpretation of results for each sample.

14. **Supercritical fluid extraction and chromatography** (SFE and SFC) (31,61) are the choices of preseparation and analytical methodology that may be used to isolate and possibly measure labile compounds that may be present in fuels and lubricants. Some sensitive compounds may be permanently altered or lost for further identification by the use of other chromatographic methods. Application of SFE/SFC methodology in the analytical scheme may provide the tools to analyze, characterize, and regulate the formation and presence of possibly harmful products. Similarly, such results may help in identifying and eventually synthesizing and using certain potent "natural inhibitors" that, thus far, have eluded isolation and identification.

    - SFC is currently being used to determine aromatic hydrocarbon content in diesel fuels.
    - If development of this technology will mature to the expected heights, it may be used in **theater level testing.**

Time requirement: 1 to 2 hours per sample.

15. **Electrochemical (EC) methods** (45) of analysis include ion-selective potentiometry, potentiometric titrations, voltametry including dc voltametry or polarography, coulometry, conductance measurements, conductometric titrations, etc. The EC methods are noted for their high sensitivity, as analyses in the picogram range are possible. However, these methods' selectivity normally is only fair. To overcome this difficulty, EC methods may be combined with chromatographic methods.

These methods provide direct tools to measure concentration of components that may undergo redox reactions. It may measure, for example, the concentration of carboxylic acids, esters, phenols, in fuels and lubricants.

If development of this technology matures to the expected heights, it may be used in **operational or higher level testing.**

Time requirement: 15 minutes to 2 hours per sample.

IV. CLASSIFICATION OF METHODOLOGIES

It is desirable to classify the various discussed methods into categories according to expected achievements such as

(a) Procedures that determine compositions of fuels and lubricants, additive ingredients, and known deterioration products,

(b) Procedures that determine compositions that, in turn, may be correlated to specific inspection property requirements and performance characteristics, and

(c) procedures that directly determine inspection property requirements and performance characteristics.

Classification Categories		
(a)	(b)	(c)
Elem. Analysis	Elem. Analysis	Elem. Analysis
FTIR and Raman	FTIR	
UV and VIS	NIR	
GC/MS	UV/VIS	
HPLC	TGA/DSC	
NMR	GC	
	GC/MS	
	NMR	

Due to the complexity of the subject, the multitude of test results, and data interpretation possibilities, modern instrumental methods resist classification into single categories. **TABLE 2** shows a cursory attempt to provide broad, multientry classification, according to the groupings discussed above. An expanded discussion of the capabilities and promises of the various methods has been discussed in earlier sections. A listing of possible correlations between instrumental analyses (identified in Section III) and several of the specification type testing required by MIL-HDBK-200G is summarized in **TABLE 2.**

Before selecting instrumental analytical techniques for POL applications, an overall assessment must be made of their potential under military environment. Such an evaluation should include the procedure's relative complexity and the risks that may be involved toward both the operators and the facility. **TABLE 3** is an attempt to summarize the attributes of the listed general instrumentations.

TABLE 2. Measurement and Correlation Possibilities

Property	Method*
Appearance	5
Ash	7
Carbon Residue	7
Cetane No.	3, 8, 9, 12
Cloud Point	3, 8
Color	5
Conductivity	2, 15
Corrosion	1, 2, 15
Distillation	8, 9
Filtration Time	Traditional, 7, 8
Flash Point	8, 9, 12
Freezing Point	3, 8, 12
FSII	2, 4
Gravity	Gravity Meter, 8
Gum	Traditional, 7, 8
Knock Rating	3, 5, 8, 9, 12
Lead	1
Metals	1
Neutralization No.	Auto. Titrator, 15
Ox. Stability	2, 7, 11, 15
Particulates	5
Pour Point	3, 8, 12
Solids	Traditional
Vapor Pressure	3, 8
Viscosity	8
Water	2, 15
Water Sep. Index	Traditional

* Numbers are keyed to methods described in Section III of text.

TABLE 3. General Assessment of Analytical Methods for Military POL Applications

Method	Potential for Applications	Method's Complexity	Safety Risks
Elem. Analysis	High	Moderate to High	Moderate
FTIR	High	Low to Moderate	Low
NIR	High	Moderate	Low
Raman	Low to Moderate	Moderate	Low
UV/VIS	High	Moderate	Low
FT-NMR	Low	High	Low
Wide-Band ^{1}H-NMR	High	Low	Low
TA	High	High	Moderate to High
GC	High	Moderate	Moderate
GC/MS	High	High	Moderate
GC/FTIR/MS	Low	High	Moderate
HPLC	Moderate	Moderate	Moderate
HPLC/^{1}H-NMR	Low	High	Moderate
SFE/SFC	Low	Moderate	Moderate to High
E. Chemical	Moderate	Moderate	Moderate

V. CHALLENGES

The challenges in modern analytical chemistry are:

(a) Define analytical chemical needs and requirements for military POL applications.

(b) Where needed, reduce complex mixtures of compounds that comprise a sample, e.g., a mobility fuel, to fractions that may (1) yield unique analytical data by the various analytical techniques, and (2) use the combined analytical results to arrive at reasoned conclusions about the sample.

(c) Correlate the analytical conclusions with performance parameters that characterize the sample.

The following is a brief summary of some of these considerations:

(a) Although requirements of the present MIL-HDBK-200G may be kept and adapted to include modern analytical instrumentation, present and future studies may lead to new scopes of analytical needs as they relate to the needs of the future military commanders.

(b) To suit subsequent analytical techniques, the **complexity of the mixture may need to be reduced.** This action must be accomplished **without uncontrolled alterations,** modifications or loss of some of the compounds that comprise the sample. It has been shown that, during chromatographic manipulations of certain fuel decomposition products, some components are altered, while some other components are irreversibly adsorbed on the adsorbent column. Subsequent chemical analysis of the product, therefore, yields incomplete and questionable results.

Reduction of sample complexity must be accomplished on a quantitative basis while continually demonstrating that the results actually refer to the original sample in question.

(c) **Development of analytical chemical protocol** for the rational and expedient analysis of fuels and lubricants is necessary.

(d) **Management and interpretation of the analytical data** are becoming increasingly simplified as new generations of instrumentation gain power and sophistication.

Traditionally, the data generated by experiments or by instruments have been collected, manipulated, and interpreted by appropriately trained professionals. Now, most modern analytical instruments contain suitable computer(s) to control the operation and calibration of the instrument and help reduce the data into a useful, reportable format. These computers ease the burden on the professionals in generating increasing amounts of highly sophisticated data. The computers may (a) integrate

data from various instruments and other sources, and (b) convert raw data into information. Such information may be easily compared to computer-based specification requirements. In many instances, the computers also provide some measure of interpretation and indication of practical implications of the acquired data.

(e) **Expert systems** that not only interpret data of a single instrument, but would automatically draw well reasoned conclusions from results obtained by a battery of analytical methodologies, are still to be developed.

Development of "expert systems" requires input from several sources. Input is required from professionals who (a) develop and use these analytical methods and techniques and interpret the results of the various measurements, (b) compile, and where needed, develop additional laboratory test versus performance correlations, (c) are involved with the development of analytical instrumentation, and (d) are computer experts capable of combining the reasoning of the multitude of experts into a coherent computer program.

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APPENDIX

Significance of Tests

The significance of the various tests for petroleum products is defined in several publications, including the MIL-HDBK-200G and ASTM special technical publication 7C. In the following paragraphs, brief descriptions will be given for some of the properties listed in MIL-HDBK-200G, as given in these publications:

1. **Appearance** refers to visual examination to describe workmanship and contamination in terms of free water, sediment, and suspended matter. The contaminants may plug filters and cause fuel line freezing and corrosion, etc.

2. **Ash content** of an oil is determined by burning off the organic matter and weighing the remaining inorganic materials. Ash content may indicate the presence of metal salt type additive(s), e.g., dispersants, detergents, antioxidants, and/or inorganic contamination, e.g., sand.

3. **Basic sediment and water (BS & W)** determines the presence of water and other foreign materials in burner fuels. Such components may cause fuel line malfunction.

4. **Carbon residue** relates to coking and fouling of fuel injectors by heavy fuel and lubricant components.

5. **Cetane number** defines the low-temperature starting characteristics of diesel fuels.

6. **Cloud point and pour point** define the low temperature limits of usefulness of fuels. Below these temperatures, the fuel becomes cloudy, it may not flow, and may plug filters and transfer lines due to the formation of increasing amounts of water or wax crystals.

7. **Color** of a petroleum product may be due to (a) normal color, (b) added product identifying dyes, (c) contamination, and (d) aging or fuel degradation.

8. **Conductivity** of a fuel refers to its ability to dissipate electrical charges within the fuel. Addition of conductivity additives to turbine fuels help assure that electrical charges (for example, static charges due to high-speed fueling operations) will not accumulate to cause sparks and possible explosion of the fuel tank.

9. **Corrosion** tests indicate if a fuel contains components that have tendencies to cause corrosion on certain metal surfaces, e.g., on copper or copper alloys, on silver, etc.

10. **Distillation** of a product provides a wealth of important information: it classifies the product (roughly) according to molecular weight and volatility (gasoline, turbine fuel, diesel fuel, etc). Distillation relates the fuel's flash point to the fuel's tendency to vapor lock; it may indicate the presence of low or high molecular weight contaminants. Results of distillation may also be linked to the product's heat of combustion and viscosity.

11. **Existent gum** defines the amount of nonvolatile residue present in fuels. This value may indicate the presence of some additives, the fuel's state of stability, and its tendency to form deposits.

12. **Flash point** is an indicator of a product's fire hazard. It is related to the concentration of the highest volatility flammable components within the composition.

13. **Fuel system icing inhibitor (FSII)** in fuels is usually an ethylene glycol derivative that lowers the temperature at which water crystals may form and plug filters and fuel lines.

14. **Gravity** is the ratio of the weight per unit volume of a product.

15. **Knock value or octane number** is a gasoline performance characteristic that indicates if the fuel will burn progressively by the spark-initiated flame front without preignition or uncontrolled detonation.

"Rich mixture rating" refers to the maximum performance available from a fuel, while "lean mixture rating" indicates steady state or cruising requirements.

16. **Metals** in a system may indicate the source of the crude oil, the presence of additives, or contamination.

17. **Neutralization number** is a measure of acidic components in a system that may indicate a fuel's or lubricant's tendency to corrode metals that it may contact.

18. **Oxidation stability** refers to a product's tendency to remain unchanged during prolonged storage. Unstable fuels and lubricants tend to oxidize, followed by decomposition of the oxidized species to form various deposits on induction system manifolds, valves, etc. Oxidation stability of products is increased by antioxidant additives.

19. **Thermal stability** measures the high temperature stability of gas turbine fuels. Results are indicative of the fuel's tendency to cause deposits that form when it contacts heated surfaces.

20. **Vapor pressure** measures a fuel's tendency to evaporate at a given temperature. It relates to starting, acceleration, vapor lock, and flash point of fuels.

21. **Viscosity** is a measure of a liquid's resistance to flow. A fuel's viscosity relates to the size of fuel pumps to provide adequate flow; and the configuration and dimensions of injectors to produce proper atomization, thus assuring required burning characteristics of fuels.

22. **Water** is an intolerable component of aviation fuels, as it may freeze and plug fuel filters and fuel transfer lines at the low temperatures that may be encountered during high altitude flights.

23. **Water separation index, modified (WSIM)** measures the ease with which a fuel releases dispersed or emulsified water. Low WSIM rating may indicate improper functioning of fuel filter separators. Presence of surfactants may have an adverse effect on WSIM ratings.

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